ESTECEM II

Technical Report

Ver.1.1 (2017.02.21)

Tokuyama Dental

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1.Introduction

1.1 Development Background

In recent years, simplification of handling has been promoted for resin cements, and self-adhesive resin cements (hereinafter referred to as SA cements) that require no pre-treatment of the adherends have been made available by several companies. However, the adhesion strength of SA cements is weaker than that of resin cements that require pre-treatment. Moreover, it has been reported that the water sorption and esthetic properties of SA cements deteriorate because they contain hydrophilic monomers and/or acid monomers. [1, 2]

Meanwhile, simplification of systems is also being promoted in the development of resin cements that require pre-treatment, and products have become available where the number of steps for the pre-treatment agent has been reduced or a pre-treatment agent with enhanced versatility is used. However, simultaneous achievement of both a simplified process and high adhesion has not yet been sufficiently attained. In particular, there are issues with the adhesion of resin cements when using chemical polymerization, or the adhesion of products that use one-bottle type pre-treatment agents to silica ceramics due to the risk of deterioration of the silane coupling agent.[3]

Tokuyama Dental Corp. markets "ESTECEM," which is a resin cement that enables easy and reliable adhesion and is suitable for esthetic restoration. ESTECEM is highly regarded for its adhesion strength when using chemical polymerization and its adhesion to silica ceramics, which are the issues described in the preceding paragraph. This has been achieved through the use of a two-bottle pre-treatment agent for the tooth substance "ESTELINK" and a two-bottle pre-treatment agent for prosthetic materials "TOKUYAMA UNIVERSAL PRIMER".[4, 5, 6] In addition, the dual-cure paste "ESTECEM PASTE" has been formulated without acid monomers that cause staining to achieve better esthetic outcomes. The strength of the cured resin and its anti-staining properties have also met with high acclaim.[7, 8] However, there still remained a workability issue (ease of handling) with ESTECEM as it uses two bottles for each process of tooth surface treatment and prosthesis treatment; i.e., four bottles in total.

Therefore, Tokuyama Dental Corp. first developed "TOKUYAMA UNIVERSAL BOND," which can be applied both to the tooth substance and various prosthetic materials, followed by "ESTECEMⅡ," which has excellent workability (ease of handling), adhesiveness, and esthetic properties, by combining TOKUYAMA UNIVERSAL BOND with ESTECEM PASTE, which has outstanding esthetic properties.

1.2 Product Description

ESTECEM Ⅱ is a dual-cure (light and/or self-cure), radiopaque, adhesive resin cement system with excellent handling, esthetic and adhesive properties to teeth and all prosthetic materials. The main components of the ESTECEM Ⅱ are the ESTECEM Ⅱ PASTE (A/B) and TOKUYAMA UNIVERSAL BOND (A/B).

TOKUYAMA UNIVERSAL BOND promotes the adhesion of ESTECEM Ⅱ PASTE to tooth structure. TOKUYAMA UNIVERSAL BOND also enhances the adhesion of ESTECEM Ⅱ PASTE to all prosthetic materials without the use of a separate primers.

ESTECEM Ⅱ PASTE has the same composition as that of ESTECEM PASTE. Therefore, ESTECEM Ⅱ has inherited the strength and esthetic properties of ESTECEM.

The characteristics of ESTECEM Ⅱ are as follows:

1. Simple handling of adhesion
2. Reliable adhesion
3. Excellent paste properties (esthetic properties, ease of handling)

*Inherited from ESTECEM PASTE.

2. Composition and Instructions

2.1 Composition

TOKUYAMA UNIVERSAL BOND contains the following: adhesive monomer new 3D-SR monomer (phosphoric acid monomer), 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), γ-methacryloxypropyl triethoxy silane (γ-MPTES), which are adhesive monomers for adhesion to tooth and various prosthetics; several monomers (HEMA, Bis-GMA, and TEGDMA) to form bonding layers; acetone, isopropyl alcohol, and water as solvents; and a borate catalyst and peroxide as polymerization initiators. *Table1.*

ESTECEM Ⅱ PASTE is the same composition as ESTECEM PASTE and contains the following: multifunctional monomers (Bis-GMA, TEGDMA, and Bis-MPEPP) as matrix monomers; silica-zirconia filler as a filler (filler loading: 74% weight); and camphorquinone and peroxide as polymerization initiators. It was designed without acid monomers that lead to discoloration for long-lasting esthetics. *Table2.*

Table1 Composition of TOKUYAMA UNIVERSAL BOND

Bond A

Basic components	Function
Phosphoric acid monomer (New 3D-SR monomer)	Adhesion for tooth Formation of bonding layer Adhesion for zirconia , alumina, and non-precious metal
MTU-6	Adhesion for precious metal
HEMA	Penetration into the tooth substance, Formation of bonding layer
Bis-GMA	Formation of bonding layer
TEGDMA	Formation of bonding layer
Acetone	Solvent

Bond B

Basic components	Function
γ-MPTES	Adhesion for glass ceramics and resin composite
Borate	Polymerization catalyst
Peroxide	Polymerization catalyst
Acetone, Isopropyl alcohol	Solvent
Water	Solvent

Table2 Composition of ESTECEM II PASTE

Paste A

Basic components	Function
Bis-GMA	Matrix Monomer
TEGDMA	Matrix Monomer
Bis-MPEPP	Matrix Monomer
Silica-Zirconia Filler	Filler (Filler Loading 74wt%)

Paste B

Basic components	Function
Bis-GMA	Matrix Monomer
TEGDMA	Matrix Monomer
Bis-MPEPP	Matrix Monomer
Silica-Zirconia Filler	Filler (Filler Loading 74wt%)
Camphorquinone	Polymerization catalyst
Peroxide	Polymerization catalyst

2.2 Indications

- Cementation of crowns, bridges, inlays, and onlays made of glass/oxide ceramics (porcelain, zirconia and alumina), metals/alloys (precious and non-precious) and resin materials including inorganic filler (composite materials).
- Repair of fractured porcelain fused to metal crowns and all ceramic restorations.
- Cementation of veneers.
- Cementation of adhesion bridges.
- Cementation of metal or resin cores, metal or glass-fiber posts.

2.3 Shades

ESTECEM Ⅱ is available in the four shades shown below.*Fig1*.

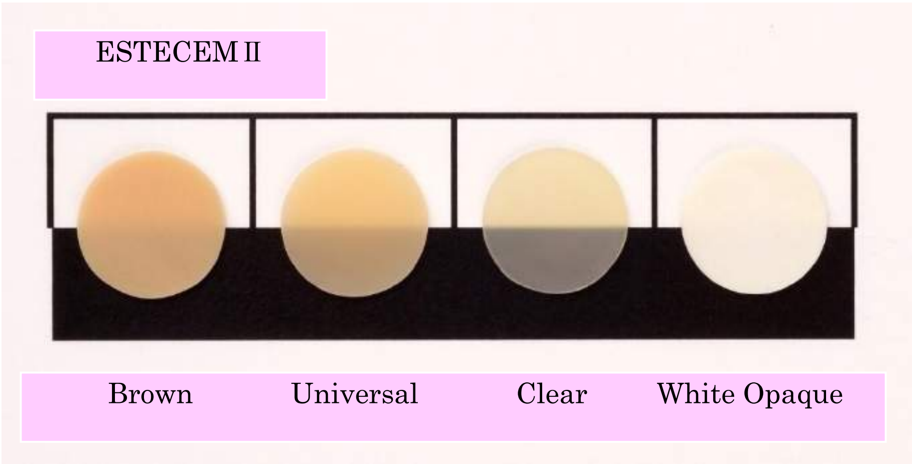


Fig1. Available in 4 shades

1) Universal:
A shade having a tooth color. Ideal for Anterior Esthetic Restorations, and generally a wide range of shade matching cases. Universal shade is included in Kit.

2) Clear:
A colorless and transparent shade. Suitable for Esthetic Crown and Veneers where the underlying tooth color provides most of the color needs.

3) Brown:
A shade having a dentin color. Suitable for ceramic or composite resin crowns.

4) White-Opaque:
A shade with high opacity. Suitable for veneer and other cases requiring masking of the underlying color.

2.4 Instructions

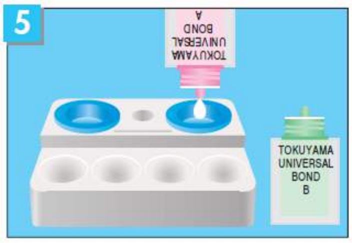
1.Tooth Preparation
Remove contamination from the tooth surface such as plaque, temporary cement residue, oil from materials to test crown fit, oil mist from a hand-piece, saliva, blood and exudate fluids by brushing with flour pumice, ultrasonic scaling or cleaning the surface of the tooth with alcohol. Thoroughly rinse and dry the surface.

Etching of the tooth structure (optional)
Clinically adequate adhesive bonds are achieved by the application of the adhesive. Selectively etching the enamel or using the "total etching procedure" can achieve higher adhesive values.
(a.) Selective-enamel-etch technique
Apply phosphoric acid gel only to the enamel surrounding the margin of prepared cavity and leave phosphoric acid gel in place for 10-15 seconds. Rinse the etched surface thoroughly (at least 15 seconds) with water, and then dry with mild air.
(b.) Total etch technique
Apply phosphoric acid gel onto the prepared enamel first, and then the dentin. The etchant should be left to react on the enamel and dentin for 10-15 seconds. Rinse thoroughly (at least 15 seconds) with water and dry with mild air or with cotton pellets; do not overdry.

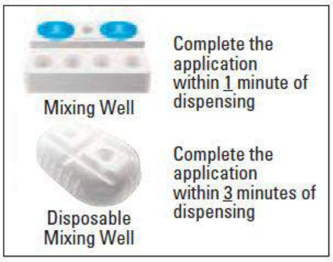
2.Restoration Preparation
Follow the procedure shown below, depending on the material to be restored.
1) Ceramics other than Porcelain, and Composite materials
Roughen the interior of the restoration by sandblasting (0.1 to 0.2MPa), air abrasion or grinding with a diamond bur using a slow speed to prepare the surface for adhesion.
2) Porcelain
DO NOT roughen the surface; Prepare the interior of the restoration, providing a clean and fresh surface for adhesion.
3) Metal restorations
Roughen the area by sandblasting (0.3 to 0.5MPa), air abrasion or grinding with a diamond bur using a slow speed to prepare the surface for adhesion.

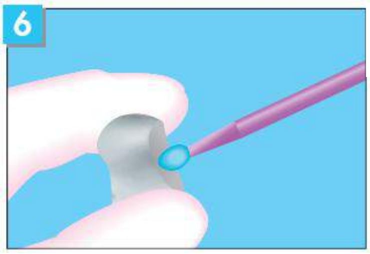
3.Thoroughly rinse the surface

4.Dry the surface



5. Restoration and tooth pretreatment with the TOKUYAMA UNIVERSAL BOND
Dispense one drop each of TOKUYAMA UNIVERSAL BOND A and B into the mixing well or disposable mixing well and mix



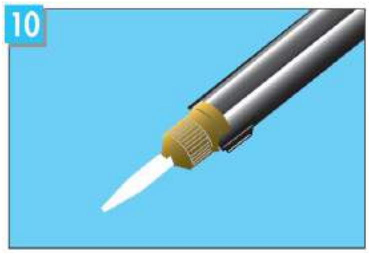


6. Apply the mixed bond on the surface of the restoration to be bonded.

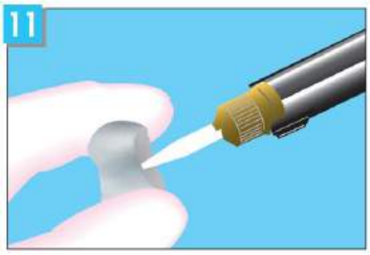
7. Apply mild air to the surface.

8. Apply the mixed bond on the surface of the tooth to be bonded.

9. Apply weak air continuously to the surface until the runny bond stays in the same position without any movement, then mild air to the surface.



10. Cementation and Final Bonding
Attach a mixing tip to the syringe according to " How to attach the mixing tip ".



11. Apply the mixed PASTE to the surface of the restoration and place the restoration on the tooth with firm pressure.

12. Removal of excess PASTE

- When using the light-cure method: Light-cure the excess PASTE with a dental curing light for 2 to 4 seconds and remove the gel state of the excess PASTE.
- When using the self-cure method: Remove the gel state of the excess PASTE within 1 to 3.5 minutes after seating.

13. Final PASTE hardening by light curing

- In case of translucent restoration materials such as ceramics: Irradiate the light to the applied PASTE on the seated restoration for 20 seconds or more.
- In case of non translucent restoration materials such as metal: Light-cure along margins for 20 seconds or more, then allow the PASTE to set for 8 minutes.

*If light curing is not enough, there is a possibility of poor adhesiveness.

2.5 Adhesion Mechanism

TOKUYAMA UNIVERSAL BOND enables reliable adhesion to the tooth due to the adoption of 3D-SR technology using a new 3D-SR monomer, and BoSE technology using a borate initiator.

TOKUYAMA UNIVERSAL BOND also contains the following adhesion monomers that are effective for a range of prosthetics: new 3D-SR monomer (for adhesion to non-precious metal, zirconia, and alumina), MTU-6 (for adhesion to precious metals), and γ-MPTES (for adhesion to glass ceramics and resin composite). New 3D-SR monomer has effect to adhesion on both tooth and some prosthetics (non-precious metal, zirconia and alumina).

Therefore, TOKUYAMA UNIVERSAL BOND adheres both to tooth and prosthetic materials without the need for any additional primer.

The mechanism of adhesion to tooth and each prosthetic is described in detail below.

2.5.1 Mechanism of adhesion to tooth substance

Tokuyama Dental Corp. developed 3D-SR technology to improve adhesion of the bonding agent Tokuyama Bond Force to the tooth substance[9, 10]. This 3D-SR monomer (1st generation), having several functional groups that can interact with calcium and polymerizing groups per molecule *Fig.2*, interacts with calcium in the tooth substance at multiple points to create strong adhesion to the tooth structure surface. Further, three-dimensional cross-linking occurs via calcium, and copolymerization between adhesive 3D-SR monomers and other monomers contributes to the formation of a very strong bonding layer. *Fig.3.*

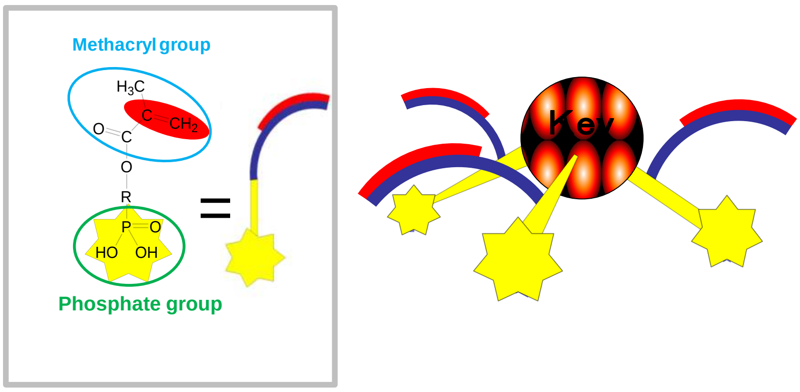


Fig.2 1st Generation 3D-SR Monom

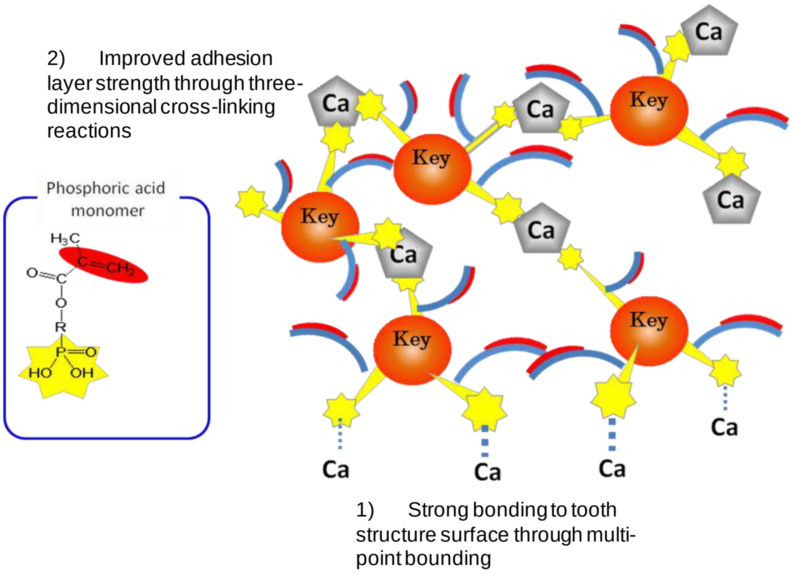


Fig.3 Three-dimensional cross-linking reactions of adhesive SR monomers and calcium ions

To achieve the enhanced properties, our patented 3D-SR monomers were improved and the chemistry was optimized (2nd generation, *Fig.4*). In comparison to the previous Bond Force generation, the number of groups interacting with calcium groups and polymerizing groups per molecule of new 3D-SR monomer has been successfully increased. With the new and improved 3D-SR monomers providing a higher level of interaction with the calcium and enhanced three-dimensional cross-linking reactions, Tokuyama Bond Force II and ESTELINK provide decreased application time from the previous 20 seconds down to 10 seconds.

Furthermore, the 2nd-generation 3D-SR monomer was adopted for TOKUYAMA UNIVERSAL PRIMER as a zirconia adhesive monomer.

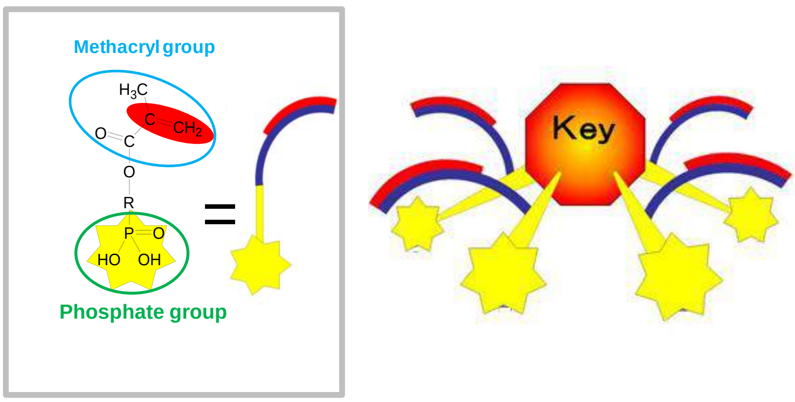


Fig.4 2nd Generation 3D-SR Monomer

TOKUYAMA UNIVERSAL BOND has an enhanced response to tooth calcium and durability by combining several phosphoric acid monomers having different chain lengths of the main chain, the alkylene group (the 3rd-generation 3D-SR monomer, *Fig.5*). This could reduce the tooth application time from 10 to 0 seconds.

Furthermore, this 3rd-generation 3D-SR monomer contributes to adhesion to non-precious metal as well as tooth, zirconia.

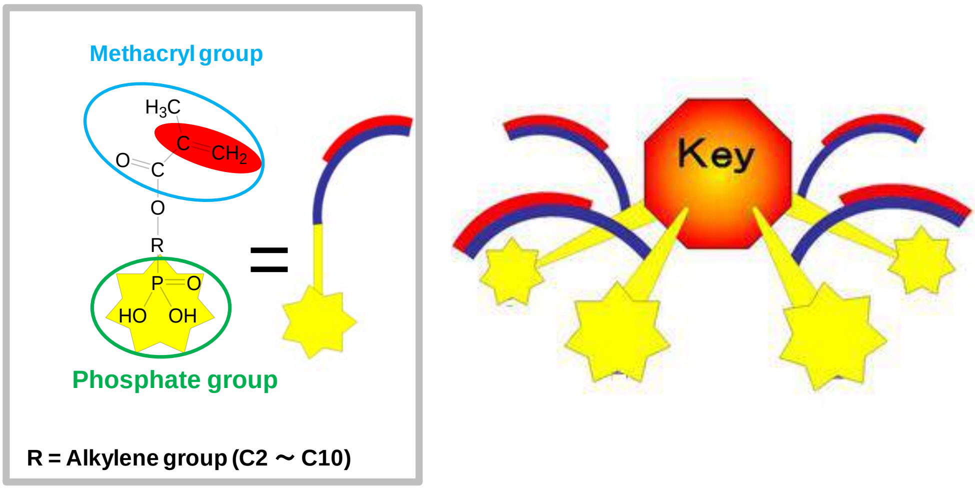


Fig.5 3rd Generation 3D-SR Monomer

2.5.2 Mechanism of adhesion to precious metal

The adhesive monomer for precious metal is MTU-6. As shown in *Fig.6*, the sulfur atom in the thiouracil group of MTU-6 interacts with precious metal (covalent bond) and additionally, the methacryl group co-polymerizes with monomers in dental-curable materials (resin cements, bonding agents, resin composites, etc.) for adhesion.

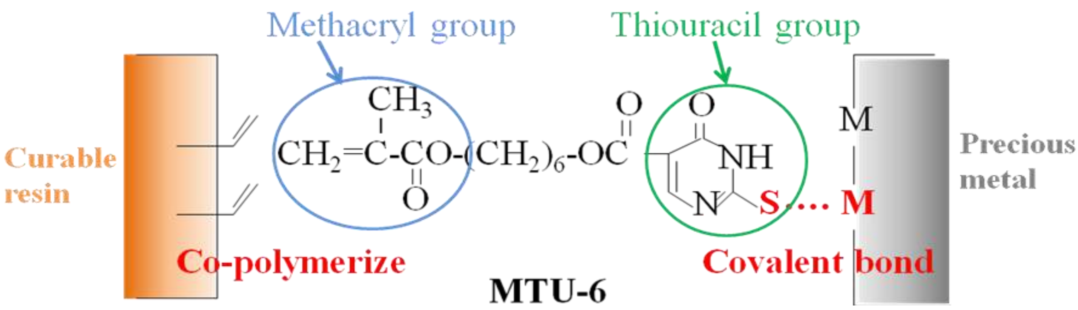


Fig.6 Mechanism of adhesion to precious metal

2.5.3 Mechanism of adhesion to non-precious metal

The adhesive monomer for non-precious metal is new 3D-SR monomer. As shown in *Fig.7*, the phosphate group of new 3D-SR monomer interacts with the oxygen atom of the passive layer of a nonprecious metal surface (hydrogen bond) and additionally, the methacryl group co-polymerizes with monomers in dental curable materials(resin cements, bonding agents, resin composites, etc.) for adhesion.

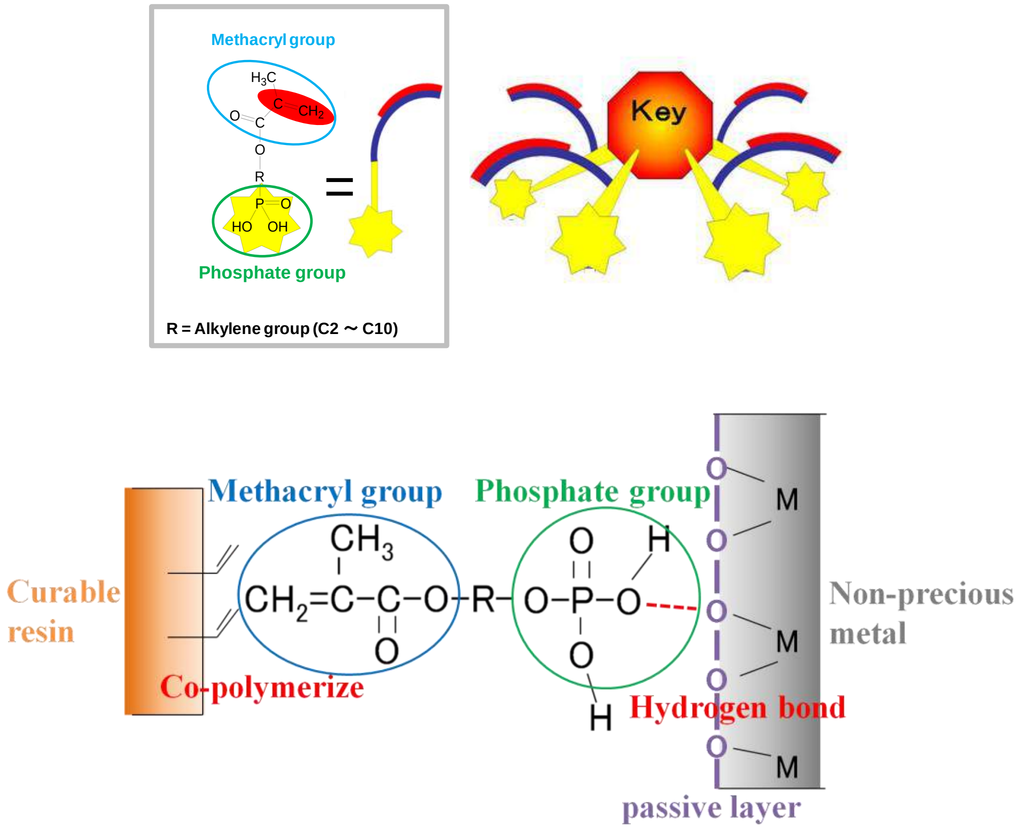


Fig.7 Mechanism of adhesion to non-precious metal

2.5.4 Mechanism of adhesion to glass-ceramics/resin

The adhesive monomer for glass-ceramics, porcelain and resin materials including inorganic filler is the new silane coupling agent, γ-MPTES. First, the alkoxy group in γ-MPTES reacts with water to form a silanol group *Fig.8* and next, a siloxane bond is formed by a dehydration and condensation reaction with the silanol group on the ceramic surface. Additionally, the methacryl group co-polymerizes with monomers in dental curable materials (resin cements, bonding agents, resin composites, etc.) for adhesion. *Fig.9.*

TOKUYAMA UNIVERSAL BOND is a two bottle-type product, and stabilization of the silane coupling agent in bottles is excellent, and the risk of deterioration of the silane coupling agent that may occur in a one bottle-type product[11] is reduced. In addition, since the new silane coupling agent, γ-MPTES is more stable in the bottle than the conventional one (γ-MPS), the adhesion effect lasts for a long time.

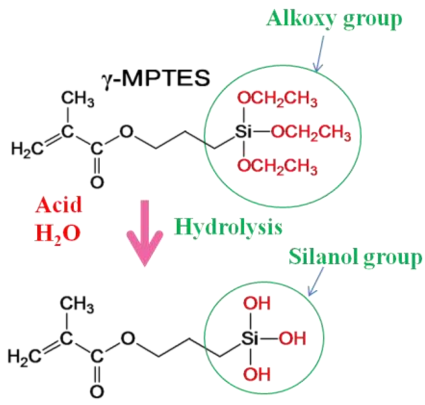


Fig.8 Hydrolysis of γ-MPTES

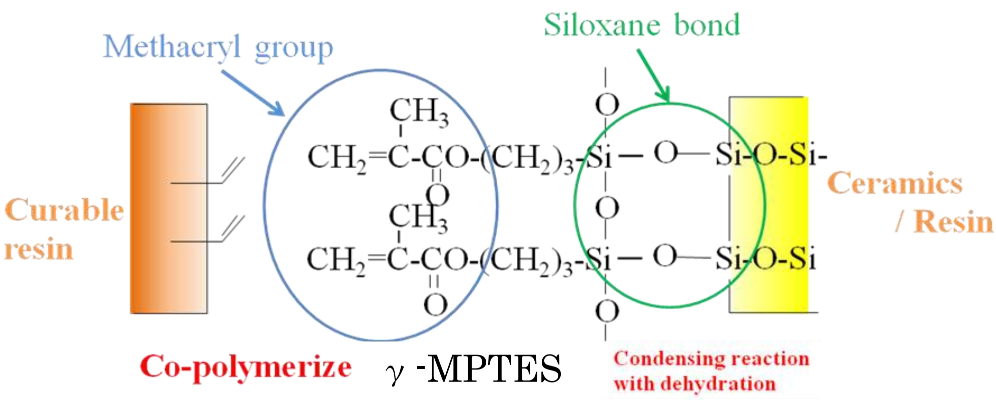


Fig.9 Mechanism of adhesion to glass-ceramics/resin

2.5.5 Mechanism of adhesion to zirconia/alumina

The adhesive monomer for zirconia/alumina is the new 3D-SR monomer (phosphoric acid monomer). It is believed that the phosphate group of new 3D-SR monomer forms chemical bonds with the zirconia/alumina surface for adhesion. *Fig.10.*

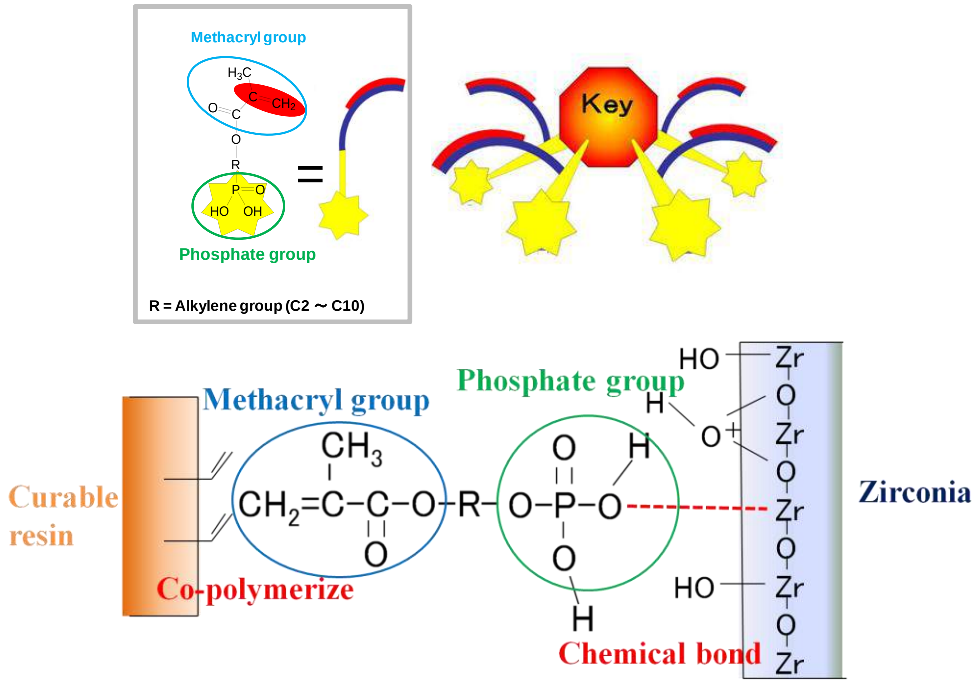


Fig.10 Mechanism of adhesion to zirconia

2.5.6 Mechanism of polymerization initiator "Contact Cure"

TOKUYAMA UNIVERSAL BOND employs our original BoSE technology with a borate initiator. The borate initiator is decomposed by acid (phosphoric acid monomer) and transformed into a borane compound which produces free radicals. In addition, TOKUYAMA UNIVERSAL BOND contains a peroxide that accelerates degradation of the borane compound and serves as a highly active chemical polymerization initiator *Fig.11*. BoSE technology is superior to the conventional chemical polymerization initiator, a benzoyl peroxide/amine system, because it exhibits high catalytic activity under strongly acidic conditions.

A thin bonding layer formed after air blow becomes hard because of rapid progression of polymerization and curing on its adhesive interface (Contact Cure), when it comes into contact with resin materials such as composite resin.

Excellent polymerization under acidic conditions made it possible to cover self-curing as well as light-curing and dual-curing type resin materials.

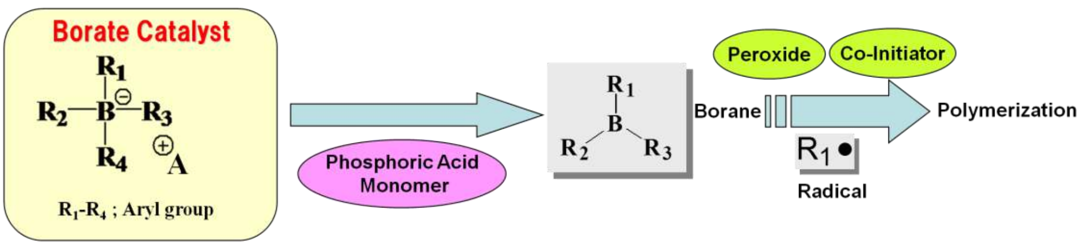


Fig.11 Mechanism of polymerization initiator

3. Features of ESTECEM II

3.1 Simple Handling of Adhesion

ESTECEM II achieves adhesion to teeth and various prosthetic materials with the same treatment method, namely, through the use of TOKUYAMA UNIVERSAL BOND. Therefore, teeth and prosthetics can be treated using the same liquid when cementing prosthetics. Moreover, even in a situation where the tooth substance and a resin core or metal core are mixed in the abutment tooth, there is no need to apply a dedicated primer for each material. ESTECEM II is a very simple system compared to competing cement systems. In addition to the above-mentioned characteristics, TOKUYAMA UNIVERSAL BOND does not require any waiting time after application, which contributes to a reduction in total chair time. *Fig.12.*

Product	ESTECEM II	ESTECEM	Panavia V5	Panavia F2.0	RelyX Ultimate	NX3	DUO-LINK UNIVERSAL	Multilink Automix	Variolink Esthetic DC
Teeth		10sec	20sec	30sec		20sec 15sec	10～15sec	30sec	20sec
Pricious metal						No treatment			
non-precious metal									
Ceramics		10sec			20sec		Note: Ceramics primer is needed for ceramics.	60sec	60sec
Indirect composite									
Zirconia									

Fig.12 Comparison of pre-treatment agents bundled with the cement systems of several different manufacturers

3.2 Reliable Adhesion

3.2.1 Adhesion of ESTECEM Ⅱ to Tooth Substance

The tensile bond strength of ESTECEM Ⅱ on enamel and dentin was measured in the self-etch and total-etch modes. The results are shown in *Fig.13*. ESTECEM Ⅱ exhibited good bonding strength during the initial stage and after thermo-cycling in both modes.

Resin Cement: ESTECEM Ⅱ (without light-cure)

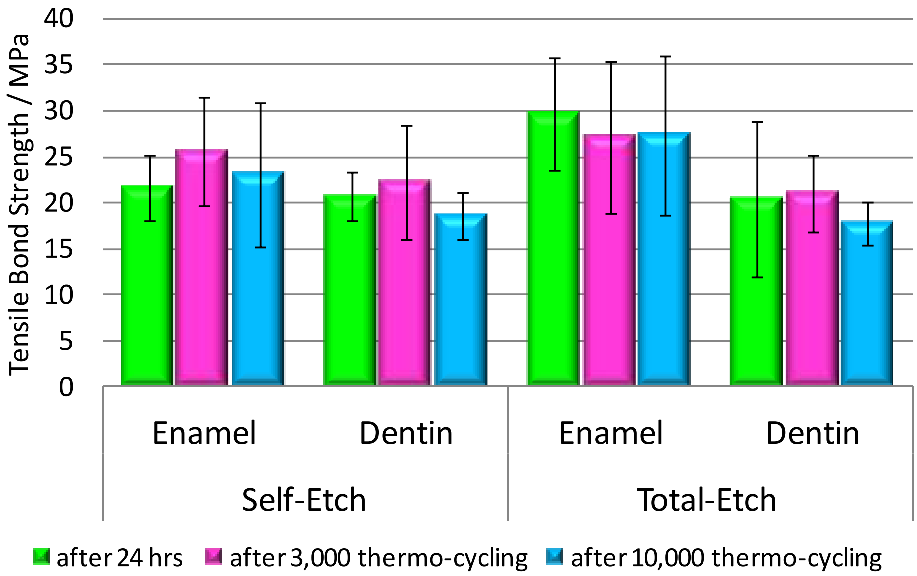


Fig.13 Tensile bond strength on enamel and dentin with varying waiting time when used with the self-etch and total-etch techniques

The tensile bond strength of ESTECEM Ⅱ and competitive products (*Table3*) to the tooth substance was evaluated (initial bond strength and bond strength after durability test). The cement was cured by chemical polymerization, based on the assumption that it may be used under light-shielding conditions. As shown in *Fig.14, 15*, the results showed that ESTECEM Ⅱ had strong adhesion to both enamel and dentin. The results also demonstrated that ESTECEM had superior durability.

Table3 Tested resin cement system

Resin Cement	Manufacturer	Abbreviation
ESTECEM II	Tokuyama Dental	ES II
ESTECEM	Tokuyama Dental	ES
Rely X Ultimate	3M ESPE	RU
NX3	Kerr	N3
Duo-Link	Bisco	DL
Multilink Automix	Ivoclar Vivadent	MA
Variolink Esthetic DC	Ivoclar Vivadent	VE
Panavia V5	Kuraray Noritake	PV
Panavia F 2.0	Kuraray Noritake	PF

Curing condition of resin cement: Self-cure (without light irradiation)

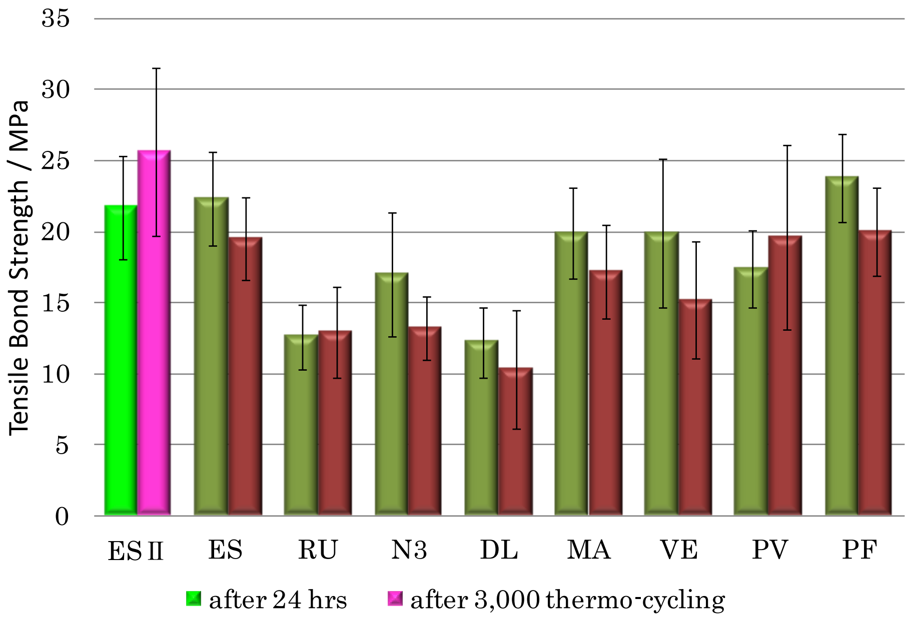


Fig.14 Tensile bond strength of resin cement systems on enamel, before and after thermo-cycling.

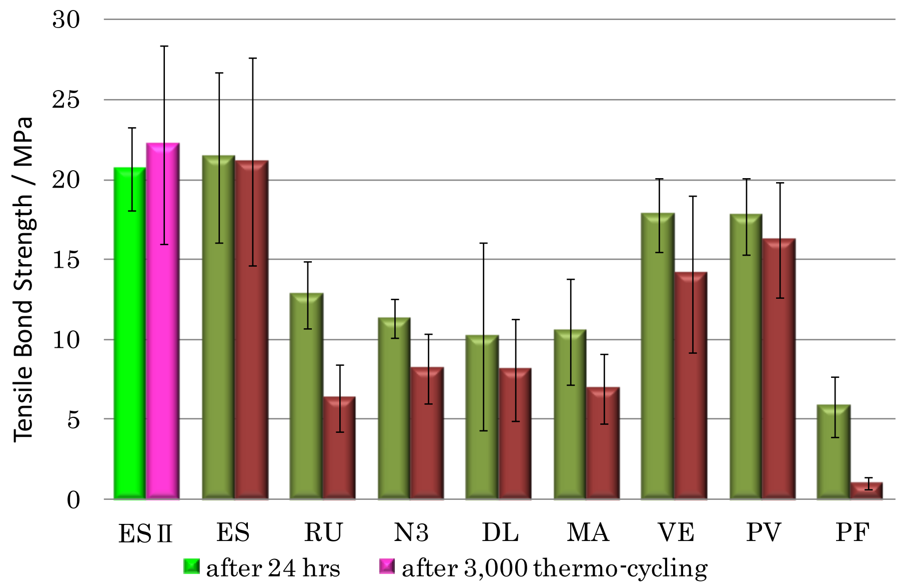


Fig.15 Tensile bond strength of resin cement systems on dentin, before and after thermo-cycling.

Fig.16 show data[12)] generated by Dr. Miyazaki at Nihon University. The data show shear bond strength at 24hours. TOKUYAMA UNIVERSAL BOND/ ESTECEM II system was compared to Sctochbond universal Adhesive / RelyX Ultimate system in the self-etch mode and total-etch mode to dentin.

TOKUYAMA UNIVERSAL BOND/ESTECEM II system exhibited good adhesion, regardless of whether phosphoric acid etching was used or light irradiation of the resin cement was performed.

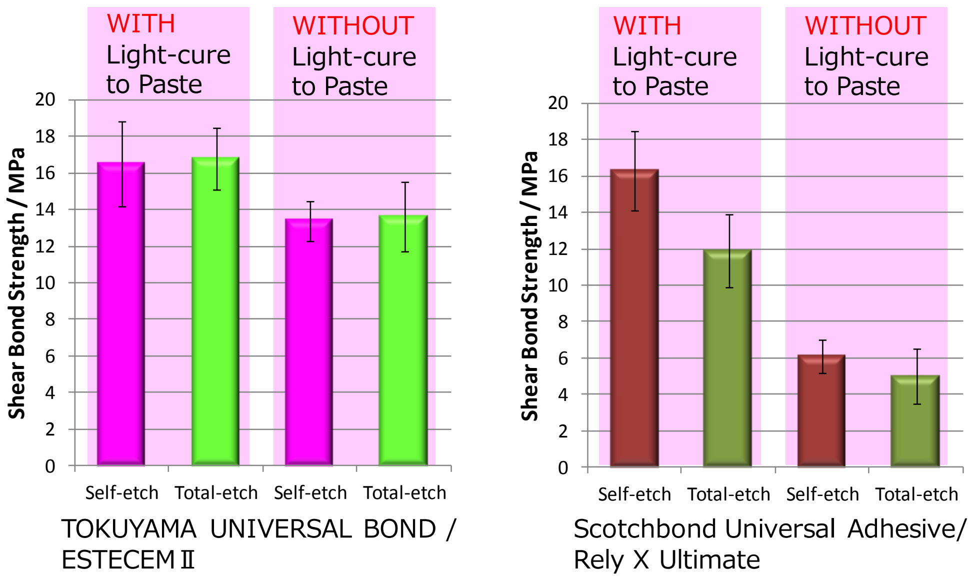


Fig.16 Shear bond strength to dentin in self-etch and total-etch technique w/wo light-curing.

Fig.17 show data[13)] generated by Dr. Sano at Hokkaido University. The data show micro-tensile bond strength at 24hours. TOKUYAMA UNIVERSAL BOND/ ESTECEM II system was compared to Scotchbond universal Adhesive / RelyX Ultimate system with/without right irradiation mode to dentin.

TOKUYAMA UNIVERSAL BOND/ESTECEM II system demonstrated good adhesion to the tooth substance even without light irradiation of the resin cement.

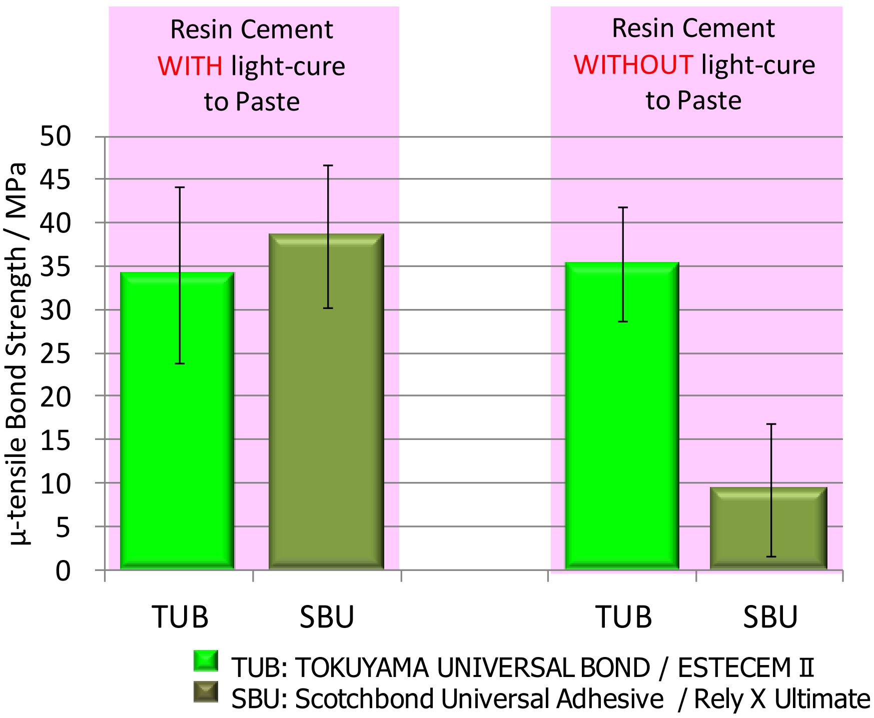


Fig.17 Micro-tensile bond strength on dentin with/without light-curing

3.2.2 Adhesion to Various Indirect Substrates

The tensile bond strength of ESTECEM II on various prosthetic materials was evaluated. The bond strengths of the prosthetic materials listed in *Table4* are shown in *Fig.18, 19*. The cement was cured through chemical polymerization, based on the assumption that it may be used under light-shielding conditions.

The results showed that ESTECEM II had strong adhesion to all prosthetic materials. These results are based on the reaction between the adhesive monomers (MTU-6, New 3D-SR monomer, γ-MPTES), which are the components of the pre-treatment agent TOKUYAMA UNIVERSAL BOND, and the prosthetics made of various materials.

Resin Cement: ESTECEM II
Curing condition: Self-cure (without light irradiation)

Table4 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name (abbr.)	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S (12S)	Au12/Pd20/Ag54/Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	GC	Casting Gold M.C. TYPEⅣ (CG4)	Au70/Pd3/Ag8/Cu16/Pt2/other1	
		Casting Gold M.C. TYPEⅢ (CG3)	Au75/Pd3/Ag5/Cu16/other1	
		Casting Gold M.C. TYPE II (CG2)	Au76/Pd2/Ag7/Cu14/other1	
	DENTSPLY SANKIN	MIRO BRIGHT (MB)	Ag72/Zn13/Sn9/In6	
		SUNSILVER CB (SS)	Ag77/Zn10/In7/Cu5	
Non-precious metal	Tokuyama Dental	ICROME (IC)	Co57.8/Cr31.6/Mo5.6/other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
		TITADENT (TI)	Ti 99.5%以上	
		TITADENT II (TI2)	Ti90/Al16/V4	
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA (SP)	—	1)Grind with #800 SiC
	Ivoclar vivadent	IPS Empress (IE)	—	
	Ivoclar vivadent	IPS e.max CAD (EC)	—	
Indirect composite	SHOHU	CERAMAGE (CE)	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	Kuraray Noritake Dental	ESTENIA C&B (ES)	—	
CAD/CAM BLOCK	VITA	Enamic (EM)	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	DENTSPLY SANKIN	KZR-CAD HR2 (KH)	—	
Zirconia	TOSO	TZ-3Y-E (TZ)	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)
	3M ESPE	LAVA Zirconia(LZ)	—	
	Kuraray Noritake Dental	KATANA Zirconia(KZ)	—	

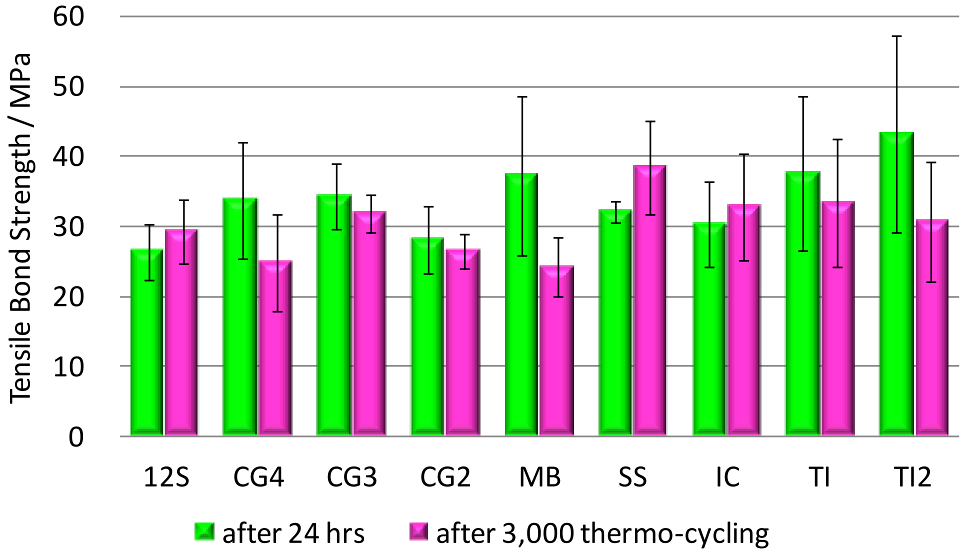


Fig.18 Tensile bond strength of ESTECEM II on metal

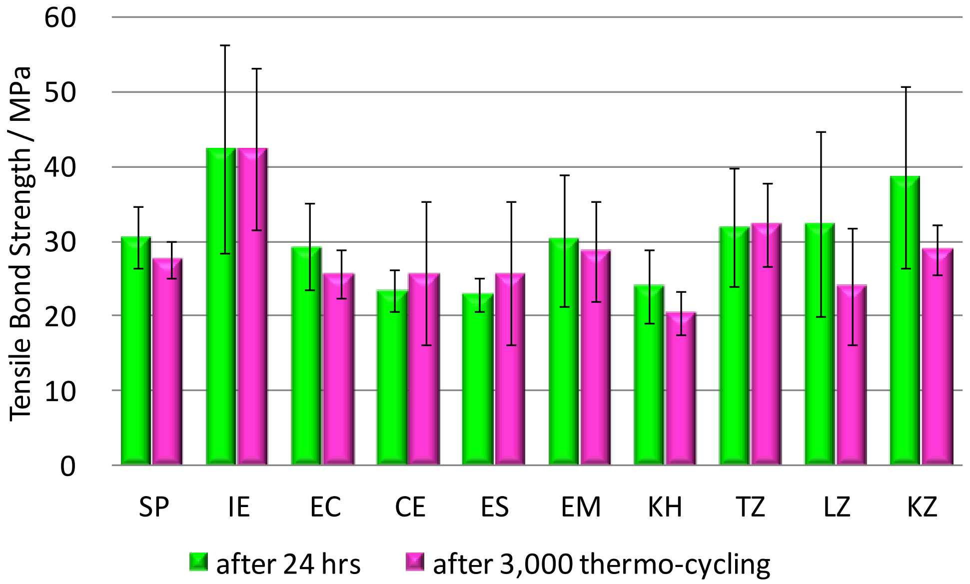


Fig.19 Tensile bond strength of ESTECEM II on ceramics, indirect resin, CAD/CAM block and zirconia

The tensile bond strength of ESTECEM II and competitive products (*Table6*) to the prosthetic materials was evaluated (initial bond strength and bond strength after durability test). The cement was cured by chemical polymerization, based on the assumption that it may be used under light-shielding conditions. As shown in *Fig.20- 24*, the results showed that

ESTECEM II had strong adhesion to all prosthetic materials. The results also demonstrated that ESTECEM II had superior durability.

Table 5 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S	Au12/Pd20/Ag54/Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Non-precious metal	Tokuyama Dental	ICROME	Co57.8/Cr31.6/Mo5.6/other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA	—	1)Grind with #800 SiC
Indirect composite	Tokuyama Dental	PEARLESTE	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Zirconia	TOSO	TZ-3Y-E	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)

Table6 Tested resin cement system for prosthetic materials

	Prosthesis				
Resin Cement (Abbr.)	Precious metal	Non-precious metal	Ceramics	Indirect composite	Zirconia / Alumina
ESTECEM II (E II)	TOKUYAMA UNIVERSAL BOND				
ESTECEM (ES)	TOKUYAMA UNIVERSAL PRIMER				
RelyX Ultimate (RU)	Scotchbond Universal Adhesive				
Multilink Automix (MA)	Monobond Plus				
Variolink Esthetic DC (VE)	Monobond Plus				
NX3 (N3)	-		Optibond XTR		
Panavia V5 (PV)	Clearfil Ceramic Primer Plus				
Panavia F2.0 (PF)	Alloy Primer		Clearfil SE Bond Primer + Clearfil Porcelain Bond Activator		
DUO-LINK (DL)	Z-Prime Plus		Porcelain primer	Z-Prime Plus	

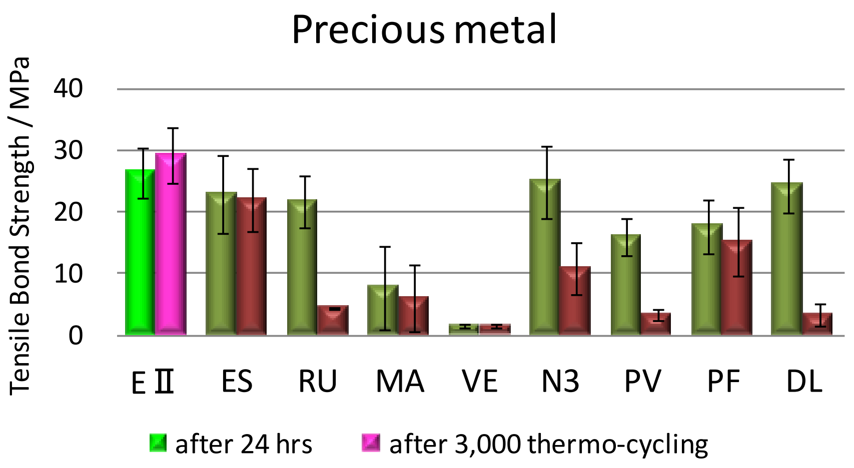


Fig.20 Tensile bond strength of resin cements on precious metal

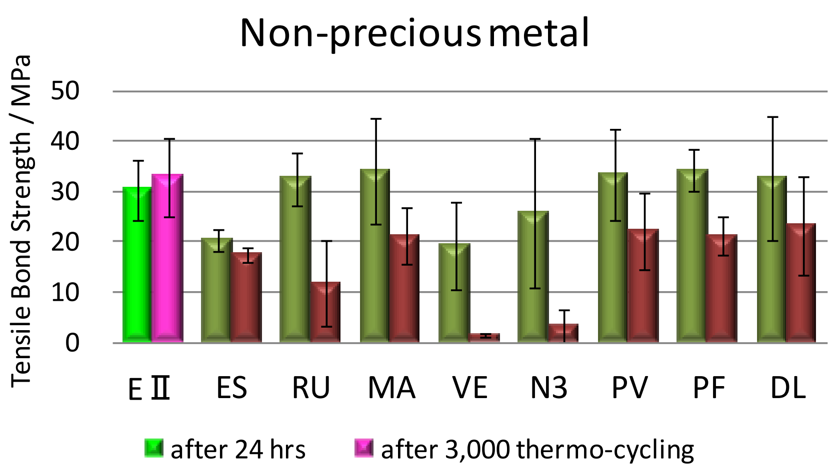


Fig.21 Tensile bond strength of resin cements on non-precious metal

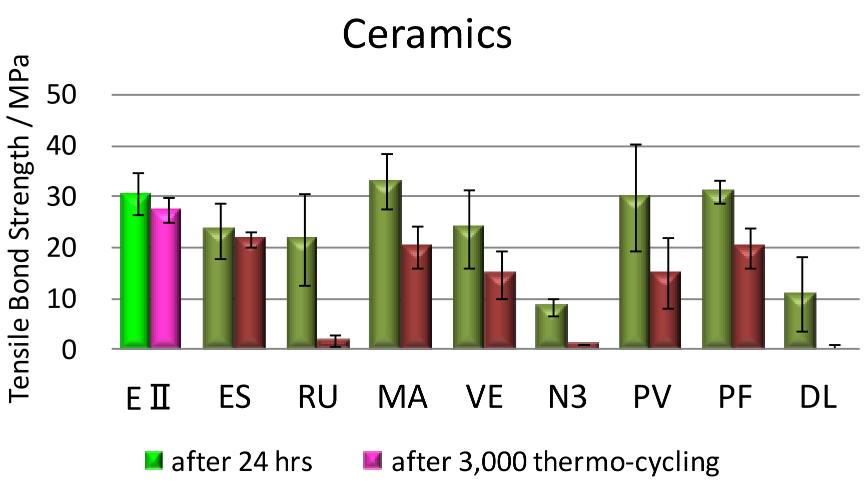


Fig.22 Tensile bond strength of resin cements on ceramics

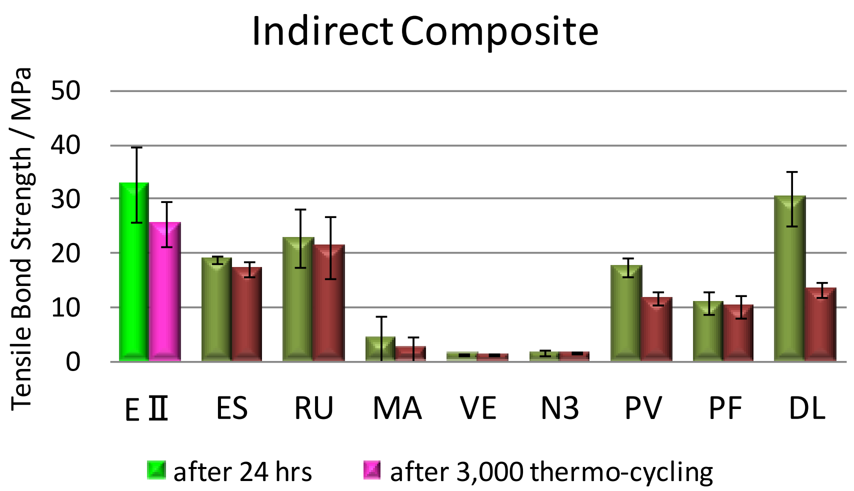


Fig.23 Tensile bond strength of resin cements on indirect composite

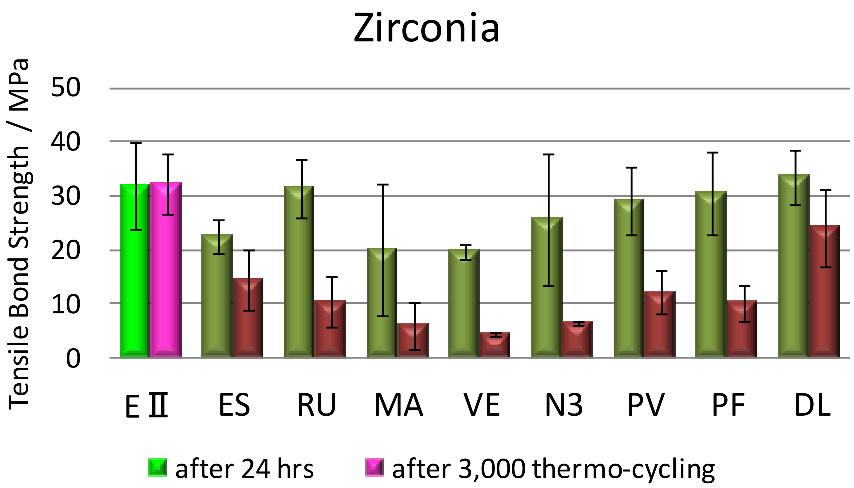


Fig.24 Tensile bond strength of resin cements on zirconia

3.3 Superior Esthetics

3.3.1 Flexural Strength and Elastic Modulus

ESTECEM II PASTE has the same composition as ESTECEM PASTE. Therefore, ESTECEM II PASTE has inherited the excellent strength and esthetic properties of ESTECEM PASTE.[7, 8)]

The flexural strength and flexural modulus of cured ESTECEM II PASTE were evaluated. *Fig.25, 26*. The evaluation method complied with ISO4049. The flexural strength and elastic modulus of ESTECEM II were at least comparable or superior to those of other manufacturers. Increasing the strength of cured paste reduces abrasion at the margin and enables restoration to achieve better esthetic results.

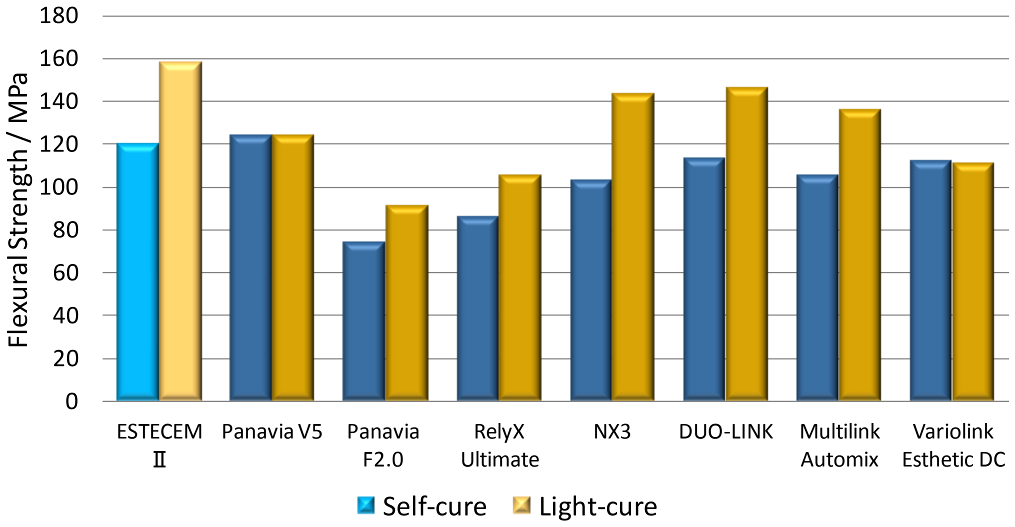


Fig.25 Flexural strength of ESTECEM II and competitive products

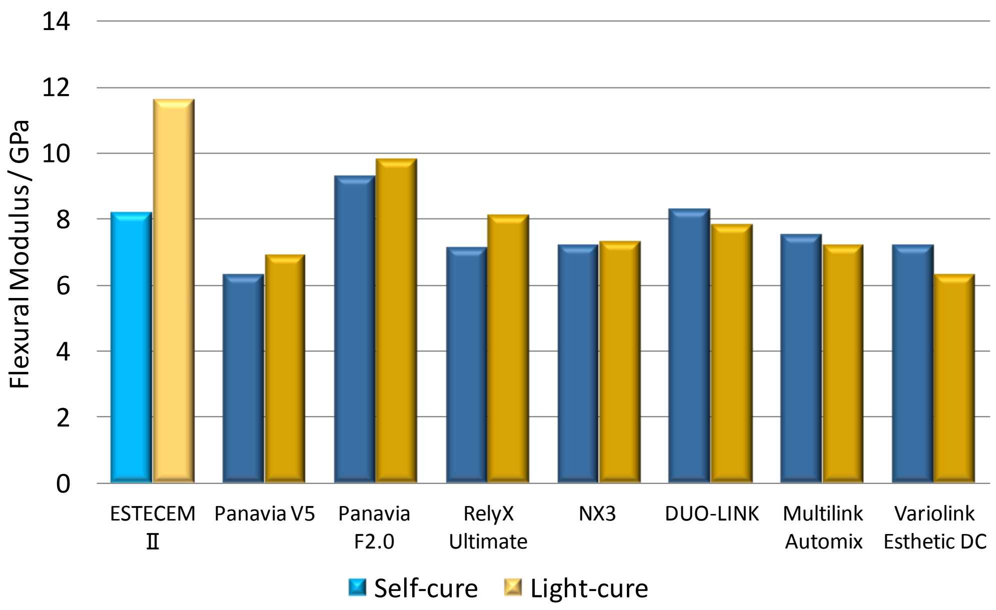


Fig.26 Flexural modulus of ESTECEM II and competitive products

3.3.2 Water sorption and the solubility

The water sorption and the solubility of cured ESTECEM II PASTE were evaluated. *Fig.27, 28*. The evaluation method complied with ISO4049. The water sorption and the solubility of ESTECEM II were both lower than the resin cements of other manufacturers, resulting in good adhesion durability, and anti-staining properties. Therefore, ESTECEM II can be expected to demonstrate reliable adhesion and produce excellent esthetic results in long-term clinical use.

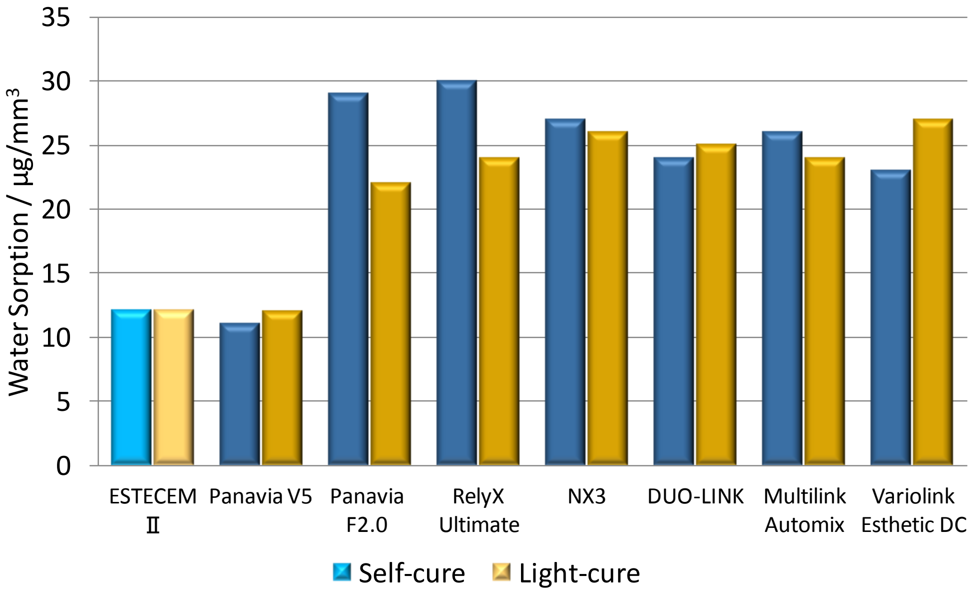


Fig.27 Water sorption of ESTECEM II and competitive products

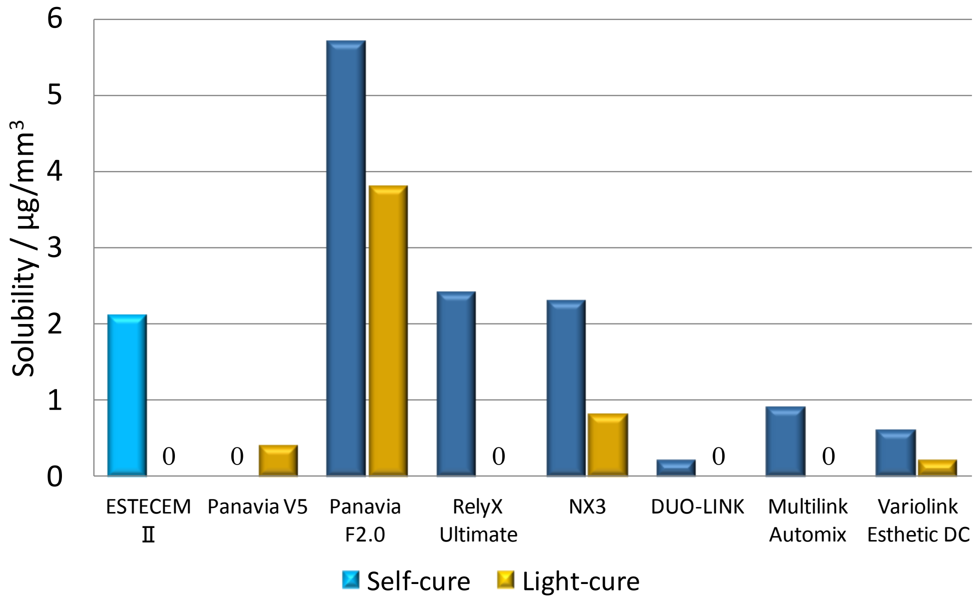


Fig.28 Solubility of ESTECEM II and competitive products

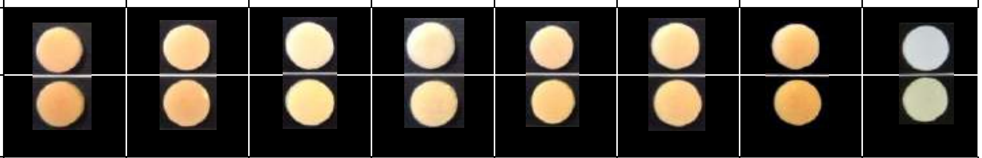
3.3.3 Color Stability

Staining tests with coffee and curry were performed for cured ESTECEM Ⅱ PASTE. *Table7, 8*. The results show that ESTECEM Ⅱ has superior anti-staining properties compared with the resin cements of other manufacturers. Therefore, ESTECEM Ⅱ can be expected to produce excellent esthetic results in long-term clinical use.

Table7 Results of staining test with coffee

	ESTECEM Ⅱ	Panavia V5	Panavia F2.0	RelyX Ultimate	NX3	DUO-LINK	Multilink Automix	Variolink Esthetic DC
Shade	UNIVERSAL	UNIVERSAL	LIGHT	A1	YELLOW	UNIVERSAL	YELLOW	LIGHT
Before test								
After test								
ΔE	4.61	12.50	11.83	8.83	8.90	8.18	10.98	19.80

Table8 Results of staining test with curry

	ESTECEM Ⅱ	Panavia V5	Panavia F2.0	RelyX Ultimate	NX3	DUO-LINK	Multilink Automix	Variolink Esthetic DC
Shade	UNIVERSAL	UNIVERSAL	LIGHT	A1	YELLOW	UNIVERSAL	YELLOW	LIGHT
Before test								
After test								
ΔE	6.55	8.87	8.40	9.48	9.70	9.56	8.94	12.80

3.4 Ease of use

3.4.1 Physical Properties Related to Cement Handling

The physical properties related to handling of ESTECEM Ⅱ PASTE and competitive products were summarized. *Table9*. The evaluation method complied with ISO4049.

Table9 Physical properties related to handling of resin cements

	ESTECEM Ⅱ	Panavia V5	Panavia F2.0	RelyX Ultimate	NX3	DUO-LINK system	Multilink Automix	Variolink Esthetic DC
Curing Time (37℃)	4'30"	2'50"	6'30"	2'40"	2'00"	2'35"	5'30"	2'15"
Working Time (25℃)	2'40"	6'00"	10'30"	4'10"	2'10"	2'00"	3'30"	3'00"
Radiopacity	YES	YES	YES	YES	YES	YES	YES	YES
Film Thickness /µm	10	8	21	7	9	7	9	17
Flowability(Vertical) /mm	0	2	2	1	0	0.5	0	0

3.4.2 Removability of excess cement

ESTECEM Ⅱ has been designed to provide "ample time for removal of excess cement." Regardless of the intensity of light used, excess cement can be removed over a prolonged period when using photopolymerization. *Table10*. It also gives sufficient time for removal with chemical polymerization. *Table11*.

Test procedure:
1. The labial side of an extracted bovine first front tooth was ground using silicon carbide paper (#600) to adjust the enamel.
2. The adjusted bovine tooth was then left to stand at 37°C under humid conditions for at least 1 hour.
3. Each cement paste (from different manufacturers) was mixed in accordance with the manufacturer’s instructions, applied to the bovine tooth, and a piece of metal 1-mm square was pressed against the paste so that the paste protruded from the sides.
4. The tooth was shielded from light at 37°C under humid conditions for a preset period of time or irradiated with light to cure the cement.
5. Cement protruding from the sides of the metal piece was removed with a explorer.

○: excess cement can be removed easily
△: cement paste is soft/hard but can nevertheless be removed
×: cement paste is too soft/hard to be removed

Table10 Removability of excess cement (using photopolymerization)

<table>
<tr><th rowspan="2"></th><th rowspan="2">Light Intensity mW/cm2</th><th colspan="12">Irradiation time / sec.</th></tr>
<tr><th>1</th><th>2</th><th>3</th><th>4</th><th>5</th><th>6</th><th>7</th><th>8</th><th>9</th><th>10</th><th>11</th><th>12</th></tr>
<tr><td rowspan="3">ESTECEM Ⅱ</td><td>200</td><td>×</td><td colspan="6">○</td><td colspan="5">×</td></tr>
<tr><td>400</td><td>×</td><td colspan="5">○</td><td colspan="6">×</td></tr>
<tr><td>800</td><td>×</td><td colspan="4">○</td><td colspan="7">×</td></tr>
<tr><td rowspan="3">Panavia V5</td><td>200</td><td colspan="2">×</td><td>△</td><td colspan="9">○</td></tr>
<tr><td>400</td><td>×</td><td>△</td><td colspan="9">○</td><td>△</td></tr>
<tr><td>800</td><td>△</td><td colspan="4">○</td><td colspan="2">△</td><td colspan="5">×</td></tr>
<tr><td rowspan="3">Panavia F2.0</td><td>200</td><td>×</td><td colspan="8">○</td><td colspan="3">×</td></tr>
<tr><td>400</td><td>×</td><td colspan="6">○</td><td colspan="5">×</td></tr>
<tr><td>800</td><td colspan="6">○</td><td colspan="6">×</td></tr>
<tr><td rowspan="3">RelyX Ultimate</td><td>200</td><td>○</td><td colspan="11">×</td></tr>
<tr><td>400</td><td>△</td><td colspan="11">×</td></tr>
<tr><td>800</td><td colspan="12">×</td></tr>
<tr><td rowspan="3">NX3</td><td>200</td><td colspan="6">○</td><td colspan="6">×</td></tr>
<tr><td>400</td><td colspan="4">○</td><td colspan="8">×</td></tr>
<tr><td>800</td><td colspan="2">○</td><td>△</td><td colspan="9">×</td></tr>
<tr><td rowspan="3">DUO-LINK</td><td>200</td><td colspan="2">○</td><td>△</td><td colspan="9">×</td></tr>
<tr><td>400</td><td>○</td><td>△</td><td colspan="10">×</td></tr>
<tr><td>800</td><td>△</td><td colspan="11">×</td></tr>
<tr><td rowspan="3">Multilink Automix</td><td>200</td><td>△</td><td>○</td><td colspan="10">×</td></tr>
<tr><td>400</td><td colspan="2">○</td><td colspan="10">×</td></tr>
<tr><td>800</td><td>○</td><td colspan="11">×</td></tr>
<tr><td rowspan="3">Variolink Esthetic DC</td><td>200</td><td>×</td><td colspan="11">○</td></tr>
<tr><td>400</td><td colspan="12">○</td></tr>
<tr><td>800</td><td colspan="12">○</td></tr>
</table>

Table11 Removability of excess cement (using chemical polymerization)

<table>
<tr><th rowspan="2"></th><th colspan="14">After placing the restoration / min.</th></tr>
<tr><th>0.5</th><th>1.0</th><th>1.5</th><th>2.0</th><th>2.5</th><th>3.0</th><th>3.5</th><th>4.0</th><th>4.5</th><th>5.0</th><th>5.5</th><th>6.0</th><th>7.0</th><th>8.0</th></tr>
<tr><td>ESTECEM Ⅱ</td><td>×</td><td colspan="7">○</td><td colspan="6">×</td></tr>
<tr><td>Panavia V5</td><td>×</td><td colspan="2">△</td><td colspan="5">○</td><td>△</td><td colspan="5">×</td></tr>
<tr><td>Panavia F2.0 with Oxygard</td><td colspan="2">×</td><td colspan="6">○</td><td colspan="6">×</td></tr>
<tr><td>RelyX Ultimate</td><td colspan="2">×</td><td>△</td><td colspan="6">○</td><td>△</td><td colspan="4">×</td></tr>
<tr><td>NX3</td><td colspan="2">○</td><td>△</td><td colspan="11">×</td></tr>
<tr><td>DUO-LINK</td><td>△</td><td colspan="3">○</td><td colspan="10">×</td></tr>
<tr><td>Multilink Automix</td><td>×</td><td>△</td><td colspan="5">○</td><td>△</td><td colspan="6">×</td></tr>
<tr><td>Variolink Esthetic DC</td><td>×</td><td>△</td><td colspan="4">○</td><td>△</td><td colspan="7">×</td></tr>
</table>

4. Conclusion

ESTECEM Ⅱ is a resin cement having the following characteristics and is a useful material for dental practice.

The development concept of ESTECEM Ⅱ is as follows:

1. Simple handling of adhesion
2. Reliable adhesion
3. Excellent paste properties (esthetic properties, ease of handling)
   *Inherited from ESTECEM PASTE.

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